DOE/MC/31174 -- 5054
(DE96000593)

Utilization of Low NO_x Coal Combustion By-Products

Quarterly Report
April - June 1995

July 1995

Work Performed Under Contract No.: DE-FC21-94MC31174

For
U.S. Department of Energy
Office of Fossil Energy
Morgantown Energy Technology Center
Morgantown, West Virginia

By
Institute of Materials Processing
Michigan Technological University
Houghton, Michigan

DISCLAIMER

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P.O. Box 880
Morgantown, West Virginia 26507-0880

By
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Michigan Technological University
1400 Townsend Drive
Houghton, Michigan 49931-1295

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UTILIZATION OF LOW NOx COAL COMBUSTION BY-PRODUCTS
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PROJECT SUMMARY - THIRD QUARTER
April 1 through June 30, 1995

TABLE OF CONTENTS

PROJECT SUMMARY

Progress on the project thus far has been very positive. Two project participants - American Electric Power (AEP) and Nevada Power Company (NPC) - are actively evaluating the use of this beneficiation technology within their systems. Representatives from both companies visited MTU and observed pilot plant runs with their ash. Representatives from IMP have traveled to AEP in Columbus, Ohio and NPC in Las Vegas, Nevada to give presentations and answer questions. In certain areas, testing has been accelerated to accommodate the companies' interests and address their needs. Both companies have had preliminary licensing discussions with MTU's Intellectual Properties Office, and both companies are investigating their own regional marketing options. In conjunction with this exciting technology transfer activity, the proposed test program is well underway and going smoothly.

TASK 1.0 - TEST PLAN

This task has been completed.

TASK 2.0 - LABORATORY CHARACTERIZATION

Task 2.1 - Sample Collection

Samples of low NOx fly ash have been received from Detroit Edison's River Rouge Unit #2 (DE2). Discussions are underway with Central Illinois Public Service Company and with AEP for a sample from one of their other plants.

Task 2.2 - Material Characterization

Material characterization of the samples from NPC, AEP, and BGE is well underway. The DE2 sample was recently received and only the particle size distribution and LOI by size has been determined for this sample to date. The size distribution and LOI by size results for the DE2 as-received sample is given on page 1 of Appendix A - Characterization Information.

Major and trace elemental analyses, including sulfur and carbon analyses, have been performed on the NPC, AEP, and BGE as-received ash samples. This information begins on page 2 of Appendix A - Characterization Information.

Major and trace elemental analyses have also been conducted on products from pilot plant Run #4 on AEP ash. This information begins on page 4 of Appendix A - Characterization Information.

Microscopic evaluation of the as-received ash samples from AEP, BGE, and NPC is underway. Some evaluation of AEP beneficiated components has been conducted Preliminary information from the Scanning Electron Microscope study of the AEP ash begins on page 6 of Appendix A - Characterization Information.

Task 2.3 - Laboratory Testing of Ash Processing Operations

Initial laboratory work has been conducted on the NPC and BGE ash samples. These were preliminary tests, using conditions and parameters developed in previous projects. The intense commercial interest on the part of AEP and NPC led to accelerated efforts in the laboratory to move the process into the pilot plant as quickly as possible. This was done both to enable AEP and NPC to see how the process worked with their ash samples and to generate clean ash and other components for subsequent product testing, particularly in the concrete subtask. The results of the BGE and NPC tests are given on page 1 of Appendix B - Laboratory Testing Information.

Currently, a laboratory flotation test program is being conducted to evaluate the effects of various processing parameters on the resulting clean ash and carbon concentrates. This program will determine the optimum parameters for the flotation process. The detailed test plan is given in Appendix B - Laboratory Testing Information, beginning on page 2. This test program will provide an in-depth study of the processing parameters. Two ash types will be studied, the AEP sample will represent Class F ash and the NPC sample will represent Class C ash. Other parameters being studied are pulp density, conditioning time, collector type and dosage, frother type and dosage, and pH adjustment. The AEP ash is currently being tested and operating and LOI data is being collected. Data analysis has not begun. This test program is approximately one half complete and will continue through the next several months.

TASK 3.0 - PILOT PLANT TESTING

Preliminary pilot plant tests (primarily to generate material for concrete testing) on AEP, NPC, and BGE samples have been conducted. Representatives from AEP and NPC were present to observe the pilot scale operations for their ash samples. A total of six pilot plant runs have been conducted on three ash samples. The compiled data sheets for each pilot plant run are given in Appendix C - Pilot Run Results.

Runs 1 through 4 were conducted on AEP ash. Run #1 was performed to shakedown the circuit and evaluate the location of the screening operation. Several modifications were made to the circuit design for Run #2, including moving the screening operation to just prior to flotation and adding two more stages of flotation for carbon cleaning. Mechanical difficulties were encountered during Run #3 and complete results were not obtained for that run. Run #4 was a repeat of Run #3. Runs #1 and #4 produced clean ash containing under 1% LOI (0.26% and 0.96%, respectively) while recovering slightly less than 60% of the initial weight of fly ash charged into the circuit. The clean ash LOI values for Runs #2 and #3 were 2.71% and 3.0%, respectively. This is on an ash that contained over 22% LOI prior to beneficiation. The carbon concentrates for all the runs were above 61% LOI, and with the exception of Run #1, the carbon concentrates were between 68.8% and 69.9% LOI.

Run #5 involved BGE ash. The clean ash LOI value was 1.3% and over 70% of the initial sample weight was recovered. A low carbon concentrate LOI value of 26.01% was obtained. Additional cleaner stages or optimization of the existing two carbon cleaner flotation stages would probably increase the LOI of the carbon concentrate.

NPC ash was tested in Run #6. Because this is a Class C ash, the % carbon was used as the primary target parameter rather than the % LOI. For comparison, the feed to the beneficiation process had a carbon content of 3.66%. The LOI determined for the dry, as-received sample was 4.0%. The pilot plant run produced a clean ash with a 1.16%C. The carbon concentrate measurements are being revised. The amount of carbon in the concentrate is far above the amount usually measured in our analyzer and this is causing difficulties and inconsistencies in the analyses for high carbon samples. Alternative analytical methods are currently under investigation.

TASK 4.0 - PRODUCT TESTING

Two product testing subtasks - Concrete Block/Brick and Activated Carbon - are not scheduled to start until Fall, 1995.

Task 4.1 - Concrete Testing

Upon receipt of the ash samples from the various utilities, a portion was set aside to be used in the production of "as-received"concrete test specimens. These sources of ash in their original form have been used as a replacement for the cement, called for in the various mixtures, to produce a concrete designed to have a 28 day strength of 3,500 psi (35S). The testing involved mixtures with a 8%, 20% and 30% cement replacement as well as a mix consisting solely of a type I cement (0%) for comparison. From each concrete mix, nine 6-inch diameter by 12-inch long specimens were prepared for compressive strength tests. Three cylinders from each mix were tested for compressive strengths following a curing period of 7 days, 28 days and 91 days in accordance with standard ASTM test methods.

As soon as clean ash was made available from each source, following a pilot plant run, the clean ash produced was also used as a replacement for the cement in the various mixtures to produce a Grade 35S concrete. As with the as-received ash the clean ash was used as a replacement for cement called for in a 35S concrete. Again mixtures involving a 0%, 8%, 20% and 30% replacement were used to produce 6" X 12" specimens for 7 day, 28 day and 91 day compressive strength tests. The clean ash was also used in mixtures to produce concrete designed to have a 28 day strength of 3,000 psi (30S) as well as mixtures designed to have a 28 day strength of 4,000 psi (40S). As with the 35S mix, these too are to be subjected to 7, 28 and 91 day compressive strength tests.

All materials used in the manufacture of the concrete tests specimens have been selected in compliance with the Michigan Department of Transportation (MDOT). The fine aggregate used met the grade 2NS specifications while the coarse aggregate met the grade 6A specifications. The size distribution of the coarse aggregate has been 4% +1", 60% -1"+0.5" and 36% -0.5" and will be maintained for the duration of the test (The size distributions are included as Table 1 and 2 in Appendix D - Concrete Testing Information). To provide the assurance required, storage of the materials being used in the phase of the study has been in a controlled environment with temperatures being held between 76° F and 90° F and the relative humidity between 30% and 40%.

The pilot plant separation for the AEP ash obtained a cleaned ash with an LOI value of 0.4%. Chemical compositions and physical properties of the as-received and cleaned AEP ash are given in Table 3 of Appendix D. The pilot plant separation for the BGE ash obtained a cleaned ash with an LOI value of 1.3. Chemical compositions and physical properties of the as-received and cleaned BGE ash are given in Table 4 of Appendix D. Similarly, the pilot plant separation for the NPC ash obtained a cleaned ash with a carbon value of 1.16%C. Chemical analysis and tests for concrete containing cleaned NPC ash have not yet been performed. No separation process has been performed yet on the DE2 ash.

For comparison, properties of as-received and the cleaned ash obtained by processing as-received ash from the Upper Peninsula Power Co.(UPPCO) and Class F ash, produced using a conventional burner, from Consumers Power (FA5) are summarized in Table 5 and Table 6 of Appendix D. The Institute of Materials Processing (IMP) studied these two ashes in a previous project funded by the Michigan Department of Natural Resources.

As received AEP ash, from a low NO_x burner, has a very high LOI or percentage of unburned carbon. The only ash the IMP has worked with previously with a similar LOI was a dust collector ash from an UPPCO facility which had a LOI value of 29.8%. Comparing Table 3 and Table 5 in Appendix D, it can be seen that the chemical composition of the as-received ash from AEP is very similar to that of the as-received dust collector ashfrom UPPCO, i.e. the total contents of silica, alumina and iron oxide are very close to 70%, and LOI values are higher than 20%. After separation, the chemical compositions of cleaned ash from both AEP and UPPCO are again very similar. The LOI value decreased significantly, the total contents of silica, alumina and iron oxide are very close to 90% and the level of iron oxide did not increase as a result of the removal of magnetic spheres. After cleaning, both ashes meet the ASTM C 618 requirements. In addition, pozzolanic tests conducted by AEP on samples of as-received and clean AEP ash showed that the clean ash met ASTM-C-618 specifications, while the as-received ash did not.

As received BGE ash, from a low NO_x burner, is similar to a Class F ash produced using a conventional burner, from Consumers Power (FA5). From Table 4 and Table 6 in Appendix D, for the as-received ash (both BGE and FA5) the total contents of silica, alumina and iron oxide are very close to 87%, and LOI values are between 6% to 7.5%. The chemical composition for cleaned BGE ash are not available yet, however, from our experience, it is expected to be very similar to that of the cleaned FA5 ash.

The compressive strength tests have only been conducted on a limited number of concrete specimens associated with AEP and BGE. The results are listed in tables 7, 9, 11 and 12 in Appendix D. Of those mixes which have been tested for their 91 day strength and have had up to 30% of the cement replaced by ash, all have exceeded the strength of the specimen containing no ash. Although we are not yet to a point where conclusions can be drawn, it is encouraging to note that all of the 28 day strengths have exceeded the design strength of 3,500 psi.

Task 4.3 - Plastic Fillers

Efforts in the plastic filler subtask have focused on determining a method to separate the fine (<5 micron) fraction of fly ash from the bulk sample and on improving the brightness of clean fly ash. After a study of equipment available on the market to classify fine material, air classification has been chosen as the means for separating the fine fraction from the parent material.

The brightness testing has indicated thus far that TiO_2 and $CaCO_3$ coatings are not effective because the crystals formed by the very slow reactions are transparent and do not improve fly ash brightness. In addition, these crystals had a tendency to not nucleate on the fly ash surface. Current effort has focused on a $ZnO/Zn(OH)_2$ coating, produced by the following reactions:

$$ZnCl_2 + 2NaOH = Zn(OH)_2 + 2NaCl$$

$$Zn(OH)_2 \xrightarrow{150^\circ F} ZnO + H_2O$$

The $ZnO/Zn(OH)_2$ coating seems easier to nucleate on the ash surface and an SEM evaluation showed that the coating appears to cover the entire particle. However, the brightness of the ash was not improved. Possible problems include contamination from fly ash impurities leaching out into the reaction solution or too thin of a coating.

TASK 5.0 - MARKET AND ECONOMIC ANALYSIS

Progress in this task has been consistent with the proposed test plan. Secondary information from the literature is nearly complete for three of the original four potential markets for fly ash component materials; concrete, ceramics and refractories, and plastic fillers. The fourth area, activated carbon, has received a modest amount of attention. Basic product description literature has been received and reviewed for general content.

The basic economic analysis of the beneficiation process has been completed with the assistance of the Denver Equipment Company and others. Operating data have been estimated based on our pilot plant experience and from US Bureau of Mines data.

The marketing and economic analysis efforts to obtain plastic resin formulations and equipment pricing and operating information have led to interesting contacts within those industries. Several companies that currently supply mineral and recycled fillers to the plastics industry have expressed an interest in fly ash. A number of equipment suppliers, including Denver Equipment Company, Dorr-Oliver, Filtra Systems, and Eimco Inc., have assisted and are offering continued assistance toward the development of an economically viable fly ash processing operation.

As mentioned previously, representatives from AEP and Nevada Power came to IMP and observed the separation of their fly ash samples. Personnel from IMP have visited both AEP and Nevada Power to give presentations and answer questions about fly ash beneficiation. Discussions with these companies about how to implement this process in their specific systems are continuing.

APPENDIX A

Characterization Information

DETROIT EDISON-2 AS-RECEIVED ASH - SIZE DISTRIBUTION and LOI by Size							
Tyler Mesh		% Weight		% LOI		Distribution	
	Wt. (g)	individual	cumulative	individual	cumulative	individual	cumulative
+65	2.0	1.68	1.68	55.52	55.52	21.64	21.64
+100	1.8	1.51	3.18	25.71	41.4	9.02	30.66
+150	3.4	2.85	6.03	10.46	26.79	6.93	37.59
+200	5.2	4.36	10.39	7.18	18.57	7.28	44.87
+270	5.4	4.52	14.91	6.77	14.99	7.13	52.00
+325	7.8	6.53	21.44	5.07	11.97	7.71	59.7
+400	4.0	3.35	24.79	5.44	11.08	4.24	63.94
-400	89.8	75.21	100.00	2.06	4.3	36.06	100.00
head	119.4			4.30			

DETROIT EDISON-2 AS-RECEIVED ASH - MICROTRAC ANALYSIS		
Channel	Cumulative	Volume
88	100.0	0.2
62	99.8	3.7
44	96.0	9.5
31	86.5	11.6
22	74.9	12.6
16	62.3	12.4
11	49.9	11.1
7.8	38.8	10.3
5.5	28.5	8.3
3.9	20.2	7.9
2.8	12.4	7.3
1.9	5.1	3.0
1.4	2.1	1.7
0.9	0.4	0.4

Table 1. Major Elemental Analyses Results for As-Received Fly Ash

Element	AEP	BGE	NP
SiO_2	44	56.64	56.93
Al_2O_3	22.35	27.77	16.31
Fe_2O_3	5.29	3.34	4.3
MgO	0.86	0.8	2.04
CaO	0.76	1.04	11.18
Na_2O	0.32	0.28	2.84
K_2O	2.35	2.25	0.72
TiO_2	1.11	1.5	0.9
P_2O_5	0.03	0.12	0.17
MnO	0.01	0.02	0.03
Cr_2O_3	0.017	0.022	0.017
Ba, ppm	1223	820	1642
Sr, ppm	978	737	1313
Zr, ppm	194	275	389
Y, ppm	63	81	32
LOI, %	23.3	6.7	4.6
SUM, %	100.75	100.76	100.53

Table 2. Trace Elemental Analyses Results for As-Received Fly Ash			
Element	AEP	BGE	NP
Ag, ppm	0.6	0.7	0.5
Al, %	11.31	14.05	8.45
As, ppm	53.7	23.2	8.4
Ba, ppm	1272	825	1539
Be, ppm	12	19	3
Bi, ppm	0.7	0.8	0.7
Ca, %	0.57	0.73	7.13
Cd, ppm	0.4	0.4	0.6
Co, ppm	34	65	7
Cr, ppm	105	149	129
Cu, ppm	141	131	67
Fe, %	4.06	2.5	3.04
Ge, ppm	1.7	0.9	0.4
Hg, ppb	145	25	545
K, %	2.05	1.89	0.54
La, ppm	71	86	44
Mg, %	0.52	0.49	1.13
Mn ppm	90	105	159
Mo, ppm	15	12	5
Na, %	0.24	0.23	2.25
Nb, ppm	21	30	15
Ni, ppm	83	120	27
P %	0.062	0.056	0.091
Pb, ppm	56	69	27
Sb, ppm	3.5	3.4	1.5
Sc, ppm	29	39	14
Se, ppm	25.4	2.2	15.9
Sn, ppm	2	2	2
Sr, ppm	975	710	1172
Te, ppm	0.2	0.2	0.2
Th, ppm	22	29	15
Ti, %	0.57	0.87	0.47
Tl, ppm	5	5	5
U, ppm	38	40	15
V, ppm	191	213	74
W, ppm	7	4	4
Y, ppm	64	81	32
Zn, ppm	90	89	54
Zr, ppm	133	196	184

Table 3. Major Elemental Analyses for AEP Run #4				
Element	**AEP-Clean Ash**	**AEP-Carbon**	**AEP-Cenospheres**	**AEP-Magnetics**
SiO_2	58.25	19.26	57.58	14.34
Al_2O_3	28.57	9.92	29.57	8.2
Fe_2O_3	5.32	0.04	3.71	77.18
MgO	1.06	0.5	1.38	0.5
CaO	0.9	0.5	0.35	0.45
Na_2O	0.43	0.05	0.38	0.04
K_2O	2.98	0.8	4.23	0.43
TiO_2	1.21	0.7	0.91	0.31
P_2O_5	0.28	0.22	0.03	0.01
MnO	0.02	0.01	0.02	0.06
Cr_2O_3	0.015	0.02	0.007	0.028
Ba, ppm	1511	712	1041	463
Sr, ppm	1129	578	432	378
Zr, ppm	278	151	191	57
Y, ppm	82	41	47	31
LOI, %	1.2	67.7	2.4	-1.4
SUM, %	100.67	99.89	100.83	100.28

Table 4. Trace Elemental Analyses For AEP Run #4 Products				
Element	Clean Ash	Carbon	Cenospheres	Magnetics
Ag, ppm	0.5	0.5	0.5	0.5
Al , %	14.82	0.48	14.8	3.82
As, ppm	35.1	82.4	13.4	42.6
Ba, ppm	1523	96	1019	426
Be, ppm	17	1	8	30
Bi, ppm	0.6	0.9	0.2	2.4
Ca, %	0.64	0.04	0.26	0.27
Cd, ppm	0.7	0.4	0.4	0.4
Co, ppm	43	3	18	95
Cr, ppm	100	12	74	161
Cu, ppm	177	11	117	168
Fe, %	4.08	0.2	2.68	49.64
Ge, ppm	1.6	2.5	0.5	1.2
Hg, ppb	15	620	95	80
K, %	2.56	0.08	3.7	0.33
La, ppm	93	4	75	24
Mg, %	0.63	0.02	0.83	0.24
Mn ppm	111	5	108	375
Mo, ppm	4	4	3	14
Na, %	0.33	0.02	0.28	0.05
Nb, ppm	25	2	17	7
Ni, ppm	99	8	43	219
P %	0.076	0.008	0.032	0.043
Pb, ppm	78	5	32	16
Sb, ppm	4.1	3.1	1.1	4.2
Sc, ppm	39	2	30	12
Se, ppm	4.5	40.4	2.8	4.2
Sn, ppm	2	2	2	2
Sr, ppm	1104	72	423	325
Te, ppm	0.2	0.2	0.2	0.3
Th, ppm	30	2	26	10
Ti, %	0.74	0.04	0.55	0.16
Tl, ppm	5	5	5	5
U, ppm	37	10	29	10
V, ppm	201	20	152	195
W, ppm	4	4	4	6
Y, ppm	86	5	48	28
Zn, ppm	103	7	35	78
Zr, ppm	194	9	162	41

Scanning Electron Microscope (SEM) Evaluation

The scanning electron microscope (SEM) produces secondary electron images (SEIs), which illustrate the morphology of particles as small as nanometers. Backscattered electron images (BEIs) give both morphological information and concentrations of heavy elements such as iron rich particles. It is because in BEIs, heavy elements, e.g., iron, will produce bright regions on the image and light elements, such as aluminum silicates will be dark, and carbon particles will be the darkest. Therefore, BEIs can give immediate qualitative information about the compositional variation of particles. The electron beam can also be positioned stationary on a particle and obtain compositional information of regions as small as microns, using energy dispersive spectroscopy (EDS). Integrated digital image analysis allowed us to analyze each particle individually.

As-received ash and the separation products from beneficiation of as-received ash were examined in the SEM for particle size range, morphology, and composition of individual particles. Each ash and ash products was examined individually. A small sample of fly ash was sprinkled from a spatula onto a strip of double adhesive carbon tape on a carbon plug. The mount was tapped gently on the side to distribute the ash particles. The mount was then carbon coated for conductivity and samples were examined in the SEM for morphological characterization. Backscattered electron images (BEIs) were obtained to show chemical variability of particles; secondary electron images (SEIs) were acquired when high resolution of surface properties was needed.

Chemical characterization of particles was accomplished through the use of energy dispersive spectroscopy (EDS) at 15 to 25 KV accelerating voltage and a current of approximately 0.2 nanoamperes. K radiation was used for all elements. The beam was directed to each particle and the resulting spectrum analyzed for Na, Mg, Al, Si, S, P, K, Ca, Fe, and Ti, calculated as oxides. Elements lighter than Na cannot be quantified by this method, detection limits are approximately 0.5 wt%. Strictly speaking, a quantitative analysis of particles with irregular surfaces is not possible since x-rays will be generated in random directions as a function of surface shape. This will cause errors in corrections made for interferences due to absorption, atomic number, and fluorescence; it will also skew the intensity measurement from the sample used in the quantitation ratio ($I_{sample}/I_{standard}$). For that reason, most of the semi-quantitative analysis performed in this study will be discussed rather than inserted into a table, to prevent a casual reader from misinterpreting the precision of the result. However, the analytical totals obtained from analysis of irregular shaped samples will give an approximation of elemental proportions in the particle, and allow us to classify the particles into compositional types.

The following descriptions were based on a combination of the above techniques.

AEP ash: A Class F Fly ash from American Electric Power, Glen Lyn Plant, Unit #6. The NO_x control is staged combustion, and the coal is a low sulfur (<1%) eastern bituminous coal.

As-received AEP ash: As-received AEP ash consisted of both amorphous particles and spheres, with amorphous particles dominating the large particles, and spheres dominating the small particles. Maximum particle size was approximately 140 micrometers. SEM photo 4033 in Figure 1 (a) is a 100X SEI which shows the typical appearance of as-received AEP ash at low magnification. Higher magnification (500X) is seen in photo 4030 in Figure 1 (b). Photo 4032 (1500X) in figure 1 (c) is a BEI which shows sphere particles in as-received AEP ash, the two bright particles are iron spheres.

The amorphous particles in the AEP ash have compositions of approximately 45 to 77 wt% SiO_2, 15 to 23 wt% Al_2O_3, and 3.0 to 4.5 wt% iron oxide, with remainder as others. The sphere particles in the AEP ash have a average composition of approximately 55 wt% SiO_2, 33 wt% Al_2O_3, and 1.5 to 3.0 wt% iron oxide, with remainder as others. The composition for the iron sphere shown in photo 4032 is approximately 69 to 88 wt% iron oxide, 7 to 20 wt% SiO_2, and 3.0 to 9.0 wt% of Al_2O_3, with remainder as others.

Separation Products from AEP Ash

Cleaned AEP ash: Similarly to as-received AEP ash, cleaned AEP ash also consisted of both amorphous particles and spheres, with spheres dominating. The maximum particle size in cleaned AEP ash was approximately 50 micrometers which is much smaller than that of as-received AEP ash. It is because a screen separation was applied to as-received AEP ash to remove coarser particles. SEM photo 4034 in Figure 2 (a) is a 100X SEI which shows the typical appearance of cleaned AEP ash at low magnification. Higher magnification (500X) is seen in photo 4035 in Figure 2 (b). No carbon particles were observed in these two images, and it is apparent that spheres are dominant in cleaned AEP ash.

Cleaned AEP ash samples were screened into six size fractions: +325M, -325M +400M, -400M +450M, -450M +500M, -500M +635M, and -635M. Each group of particles were carbon coated and analyzed by SEM with image analysis.

SEM photo 4037 in Figure 3a is a 200X BEI which shows the typical features of cleaned AEP ash in the size range between -400M to +450M (32 μm to 40 μm). Many amorphous particles with irregular shape were observed. SEM photo 4038 in Figure 3b is a 200X BEI which shows the typical features of cleaned AEP ash in the size range between -450M to +500M (25 μm to 32 μm). Again, there are a lot of irregular shaped amorphous particles, but the amount of irregular particles reduced as compared with those in the range of -400M to +450M (30 μm to 40 μm). SEM photo 4041 in Figure 4a is a 300X BEI which shows the typical features of cleaned AEP ash in the size range of -500M to +635M (20 μm to 25 μm). No significant difference in the amount of irregularly shaped particles was observed compared to the particles in the range of -450M to +500M (25 μm to 32 μm). SEM photo 4040 in Figure 4b is a 1,000X BEI which shows the typical features of cleaned AEP ash in the -635M (less than 20 μm) size fraction. Very few irregularly shaped particles were observed. Most particles are spheres with a diameter of less than 20 μm. The particles in this range may be good for application in plastic filler, painting etc.

SEM photo 4036 in Figure 5 is a 200X BEI which shows the typical features of cenospheres from processing of as-received AEP ash. The maximum particle size in the cenosphere was approximately 100 micrometers. This matched with the largest particles found in as-received AEP ash.

Cleaned AEP ash particles in different size range were mounted in epoxy, polished, carbon coated and analyzed with the SEM using ES and image analysis. SEM photo 5140 in Figure 6a is a 200X BEI which shows a typical feature of the cleaned AEP ash. It can be seen that several ash particles are semihollow, lots of small particles are contained in it. SEM photo 5155 in Figure 6b is a 600X BEI which shows a typical semihollow particle found in the +325M size particles (larger than 45 μm). It should be note that more than 100 small particles are contained in this particle which is not a cenosphere, but a +325M cleaned ash particle. The three white particles in the semihollow particles are iron rich spheres.

Future Work

Future work for characterization low NO_x ash will be performed to study the shape, composition and other characteristics of various as-received and separation products from processing these as-received ash. The results will be very valuable to help market the separation products.

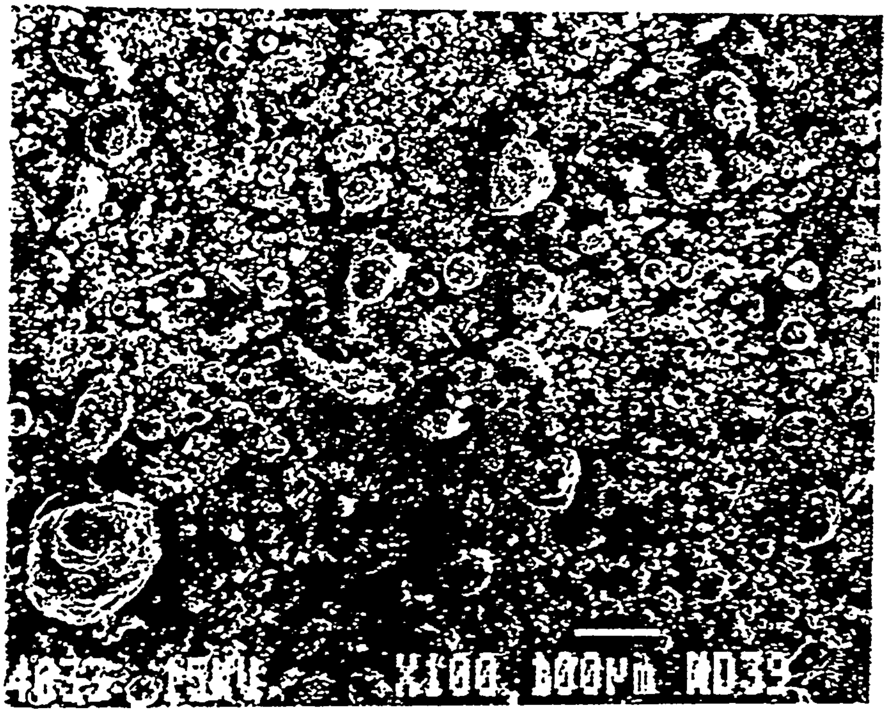


Figure 1a Typical apperance of as-received AEP ash (SEI).

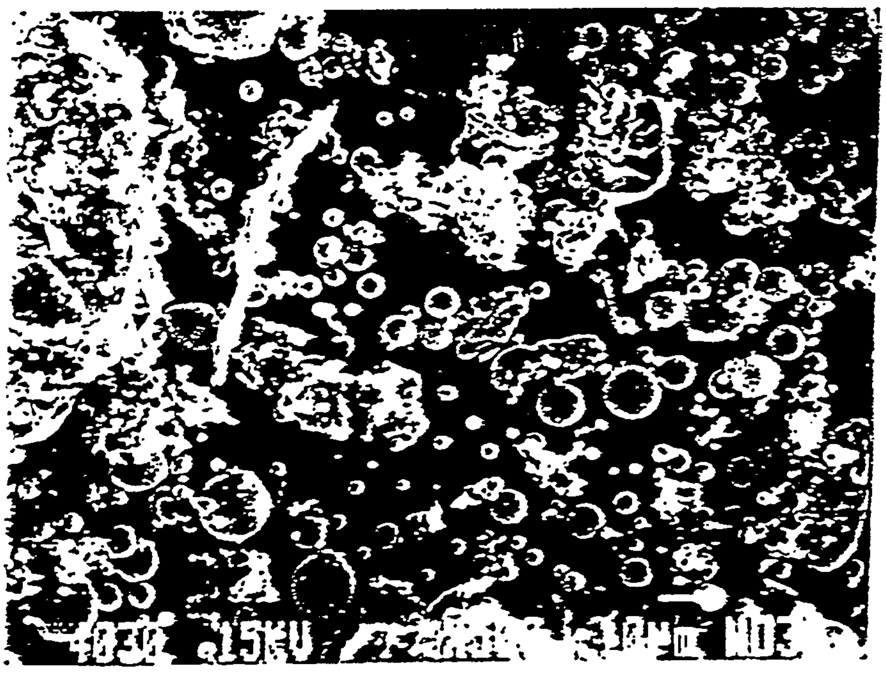


Figure 1b As-received AEp ash at high magnification (SEI)

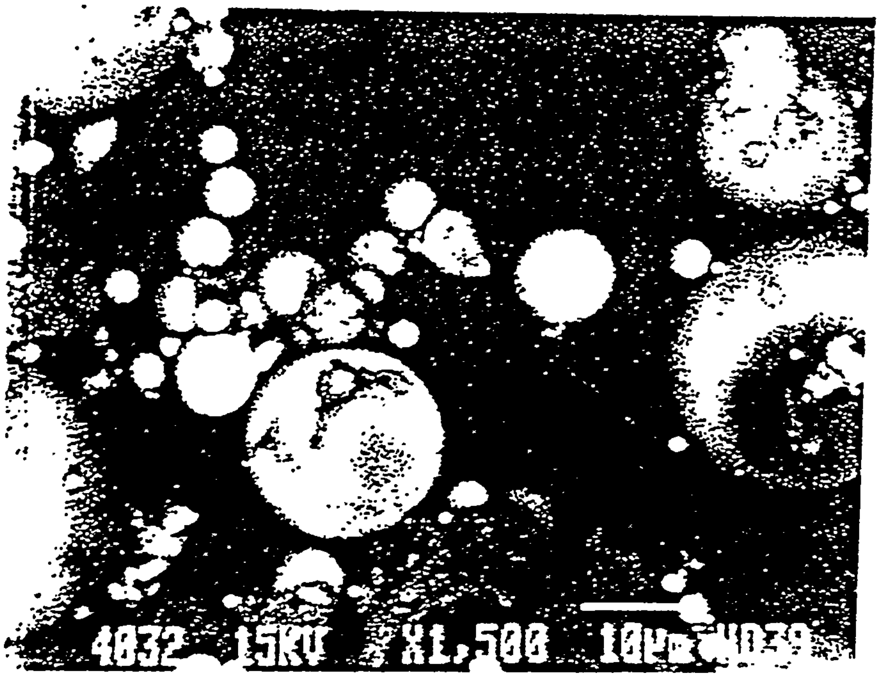


Figure 1c As-received AEp ash, two bright particles are iron spheres (BEI)

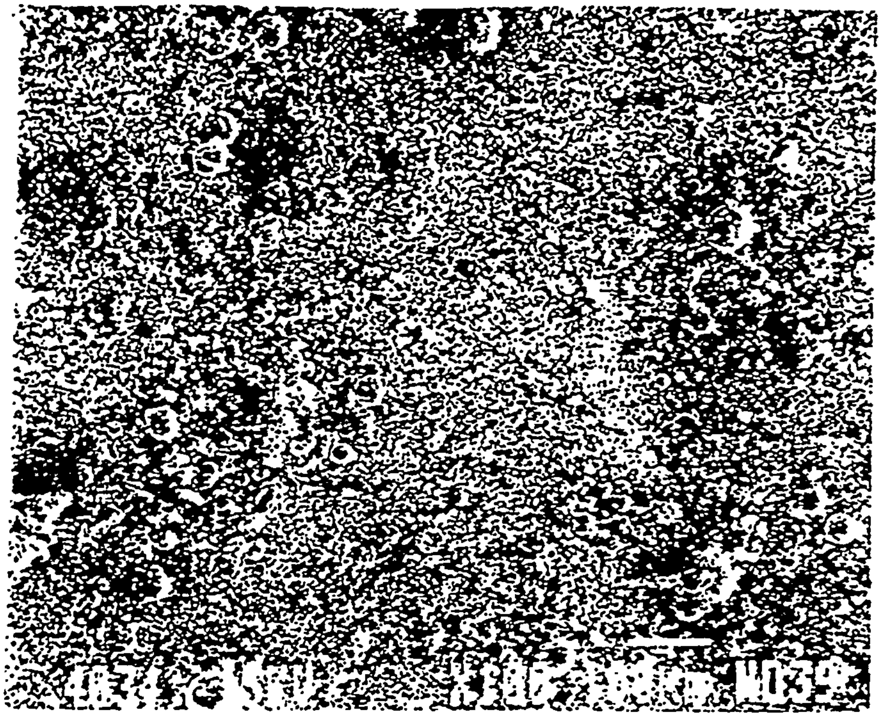


Figure 2a Typical apperance of cleaned AEP ash (SEI).

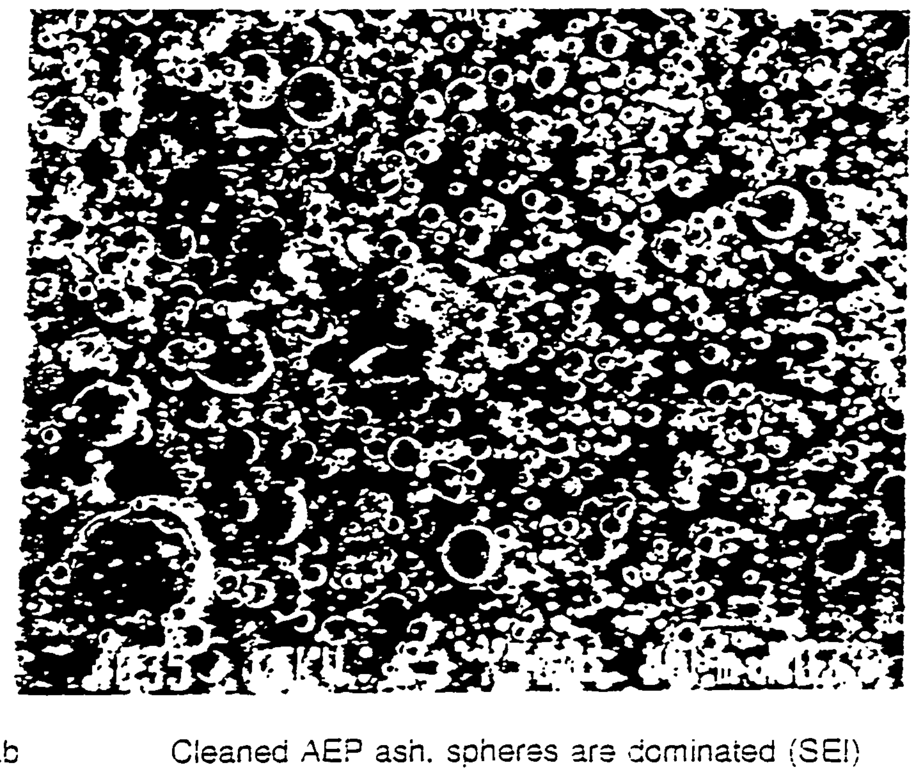


Figure 2b Cleaned AEP ash. spheres are dominated (SEI)

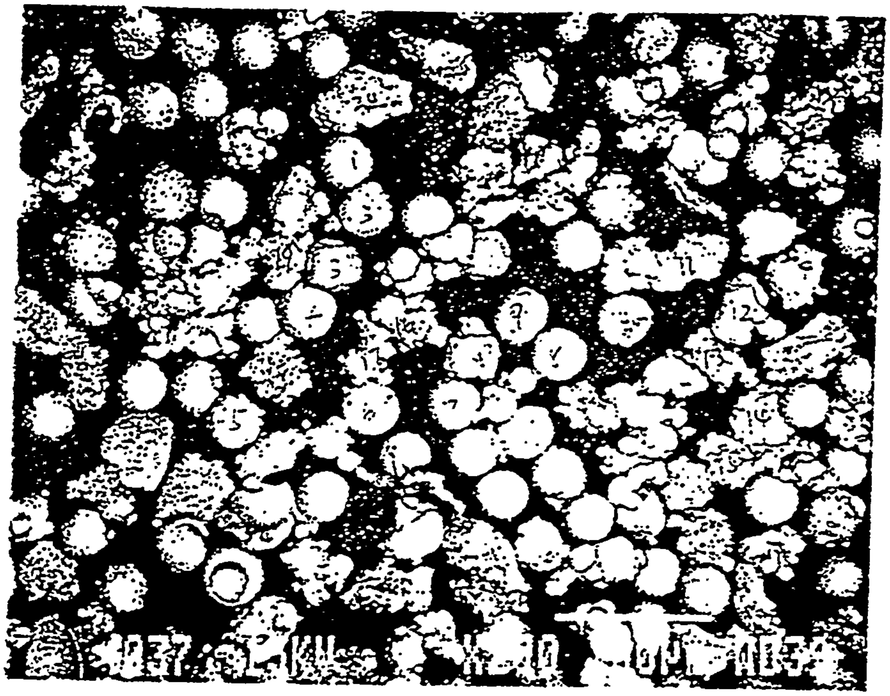


Figure 3a Cleaned AEP ash in the range of -400M + 450M (BEI).

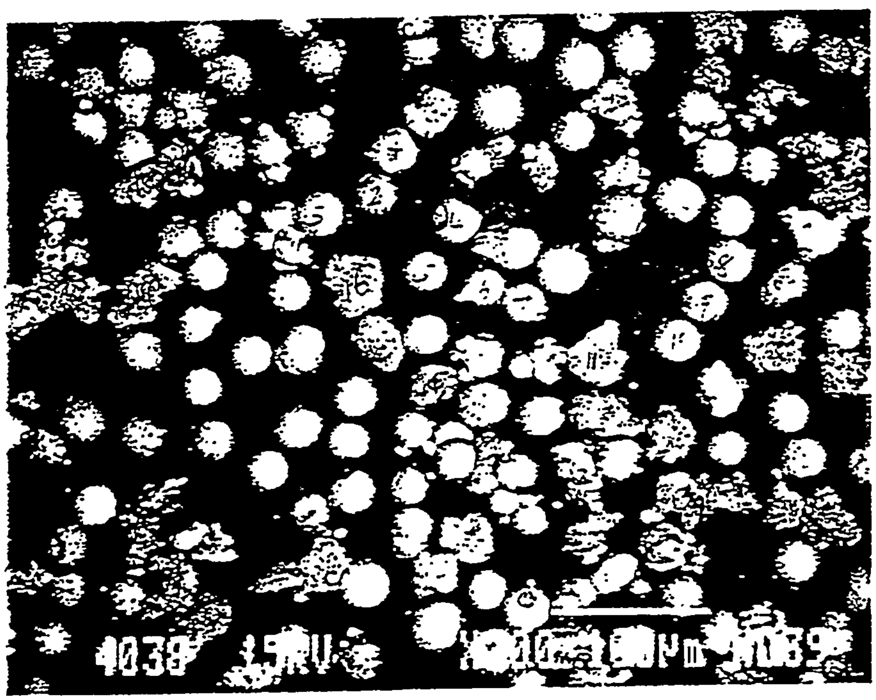


Figure 3b Cleaned AEP ash in the range of -450M + 500M (BEI).

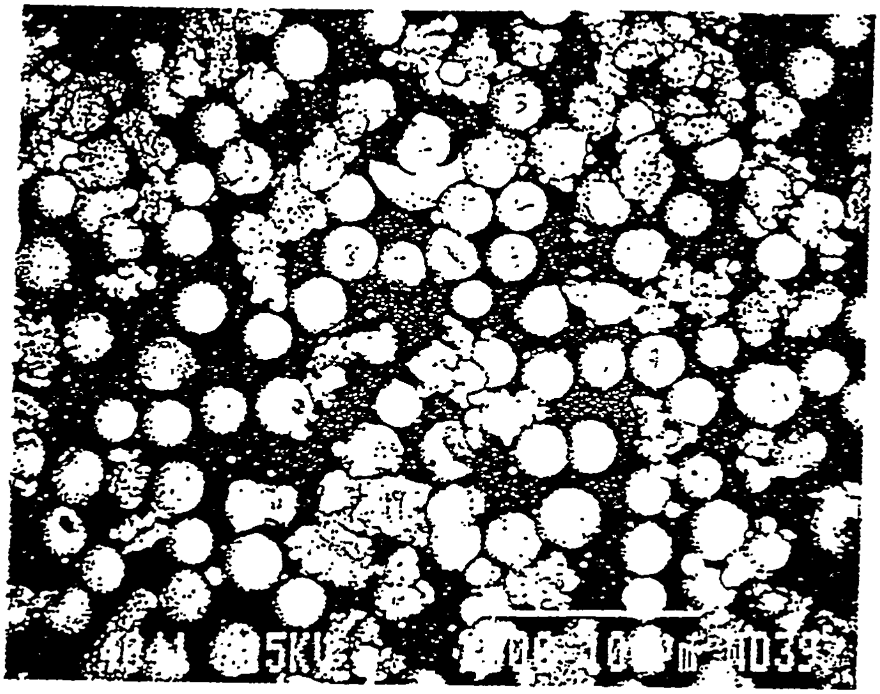


Figure 4a Cleaned AEP ash in the range of -500M + 635M (BEI).

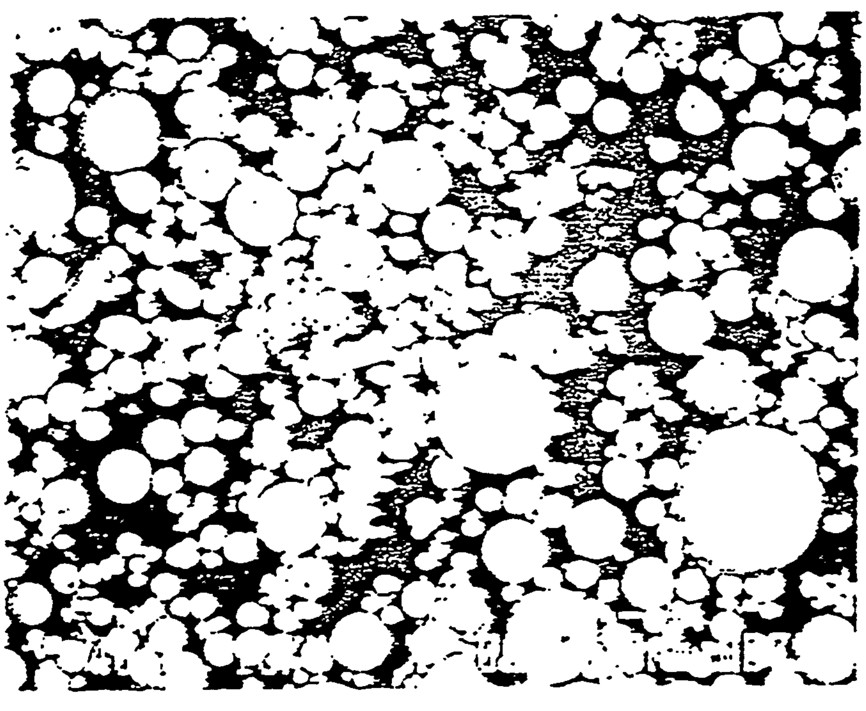


Figure 4b Cleaned AEP ash in the range of -635M (BEI).

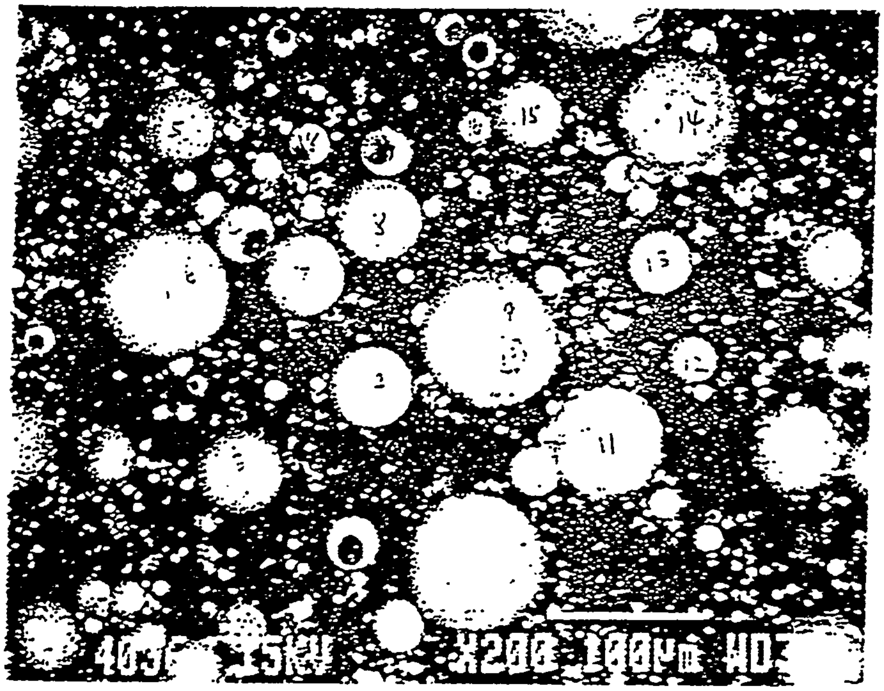


Figure 5 Typical apperance of cenospheres from Processing AEP ash

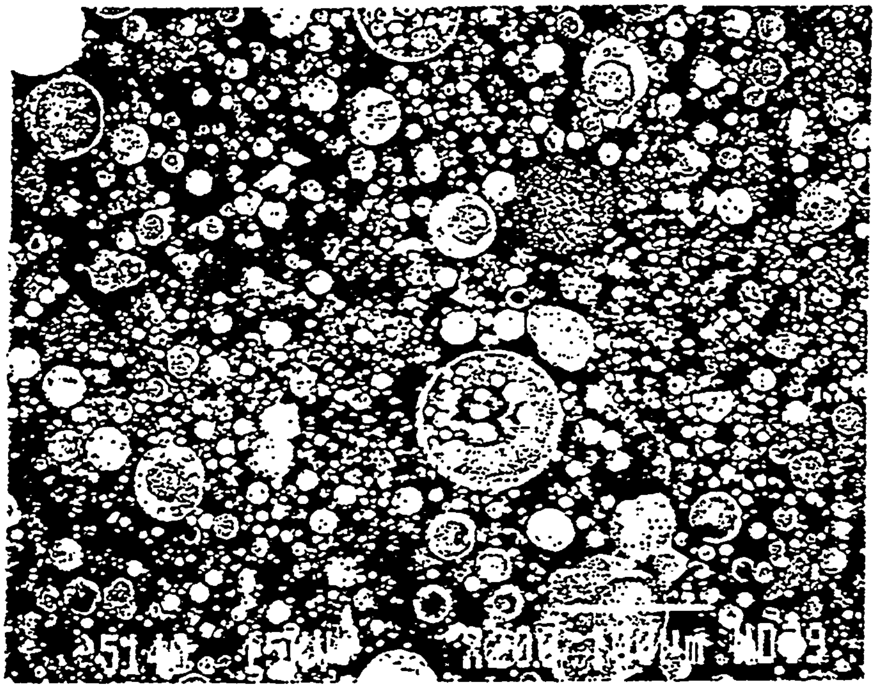


Figure 6a Cleaned AEP ash with all different sizes (-200M) (BEI).

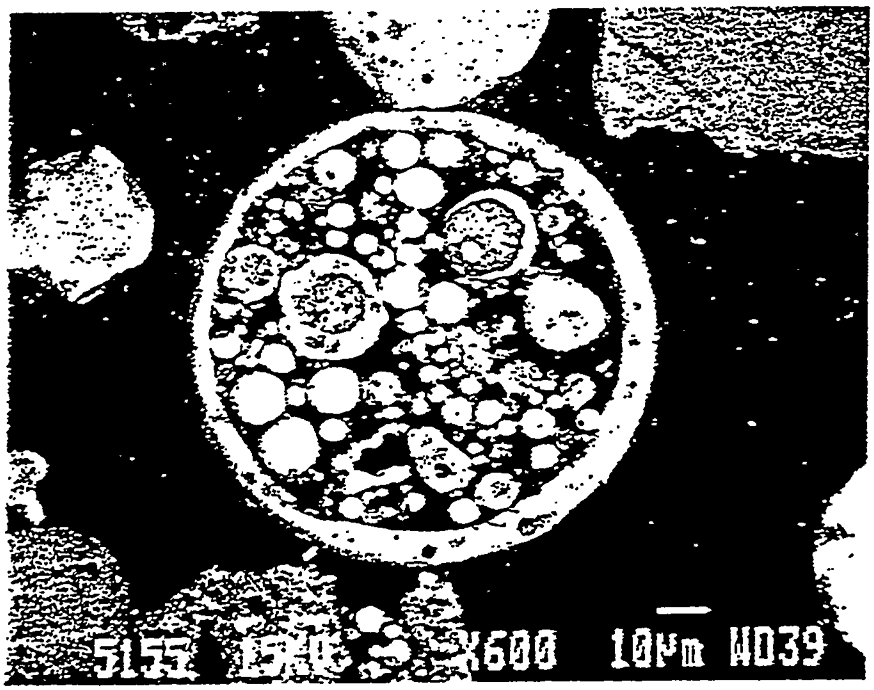


Figure 6b Typical semi-hollow particles in +325M cleaned AEP ash

APPENDIX B

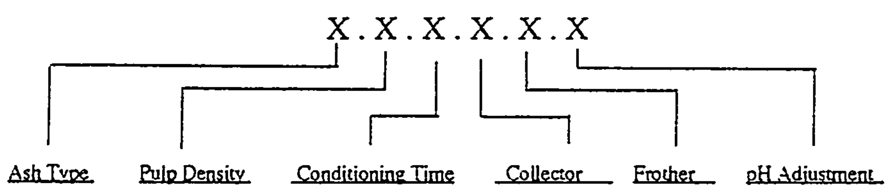
Laboratory Testing Information

SUMMARY OF LABORATORY FLOTATION RESULTS

Tes t#	Ash Type	Collector		Frother		Clean Ash		Carbon Concentrate			Screen Fraction		Middlings		Remarks
		Type	#/T	Type	#/T	%LO I	%wt rec.	%wt	%LOI	%LOI rec.	%wt	%LOI	%wt	%LOI	
29	BGE	mix	3.0	P	0.63	2.76	72.6	26.1	15.4	62.1	1.3	34.4	-	-	
30	BGE	mix	3.5	P	0.63	3.89	70.8	28.1	12.7	53.3	1.1	34.2	-	-	Repeat T29 w/ more collector
31	BGE	mix	2.5	P	0.63	3.89	72.6	26.0	15.6	49.8	1.3	36.0	-	-	Repeat T29 w/ less collector
32	BGE	mix	4.0	P	0.84	0.92	71.2	27.6	20.4	84.2	1.2	33.2	-	-	Repeat T30 w/ more collector and frother
33	BGE	mix	4.5	P	1.05	1.15	71.8	13.5	37.6	77.2	0.9	30.4	13.8	2.9	Repeat T32 w/ more reagents and 2 clnr stages
34	BGE	mix	4.5	P	1.05	1.02	70.9	10.2	49.0	77.2	0.9	30.6	18.0	3.1	Repeat T33 w/ 3 clnr stages
35	NPC	mix	1.4	P	0.63	1.36	82.7	3.6	24.8	28.8	3.3	10.4	10.3	7.1	Carbon analyses - not LOI
36	NPC	mix	1.7 5	P	0.84	0.96	80.1	16.2	9.7	59.2	3.7	8.7	-	-	Repeat T35 w/ more reagents, Carbon analyses - not LOI

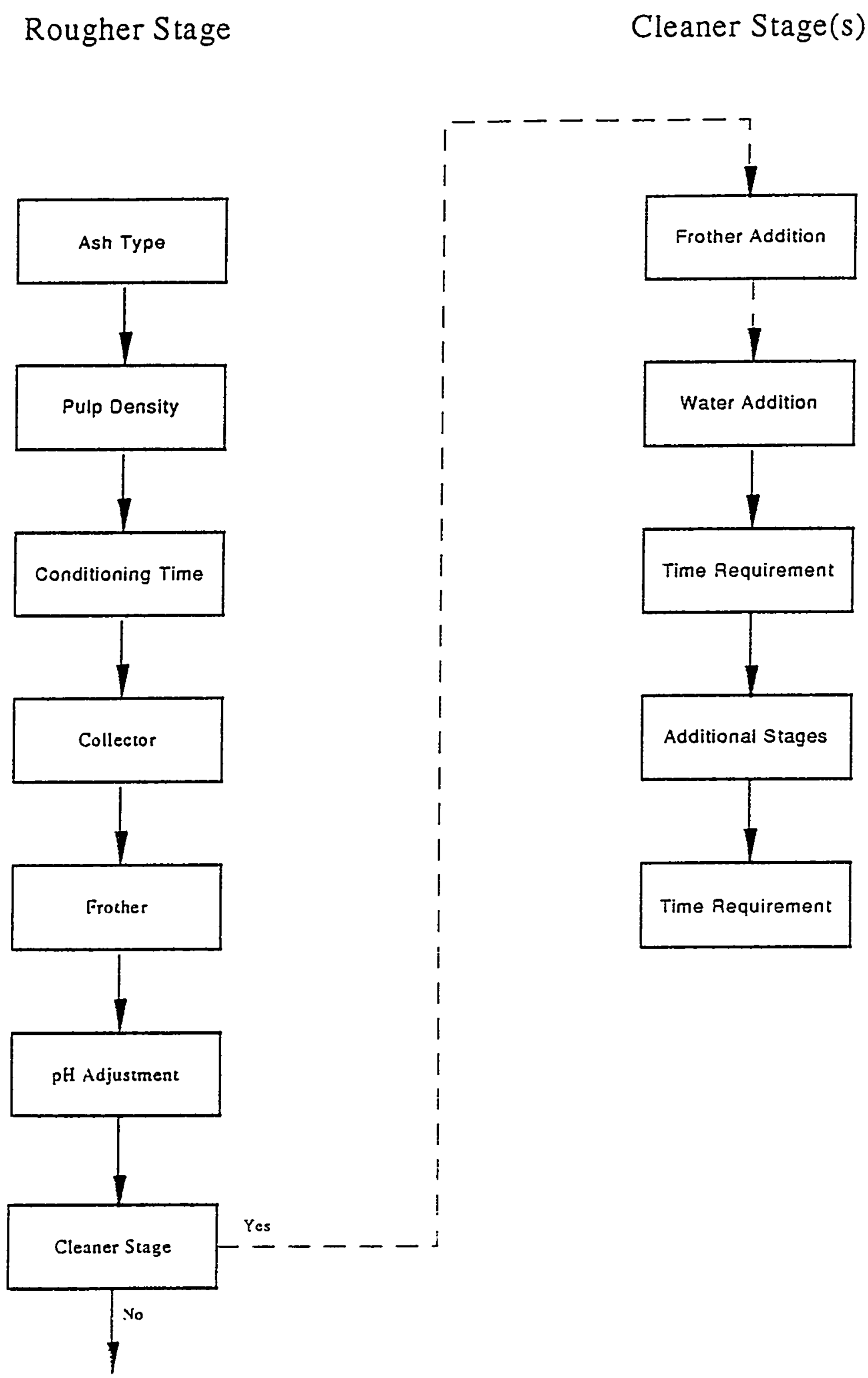
Flotation Test Plan

As a means of tracking the results from the extensive number of flotation tests to be conducted, a modified Dewey decimal classification system will be used. The format to be followed will consists of a six digit number representing each flotation test which has been carried out in the lab. The first of the six digits will indicate the type of ash being tested the second indicating the pulp density followed by conditioning time, collector type and amount, frother type and amount, whether or not a pH adjustment was made and whether or not a cleaner stage test was conducted. The results will then be tabulated correlating the individual test run with the froth LOI, Cell LOI, etc. etc.



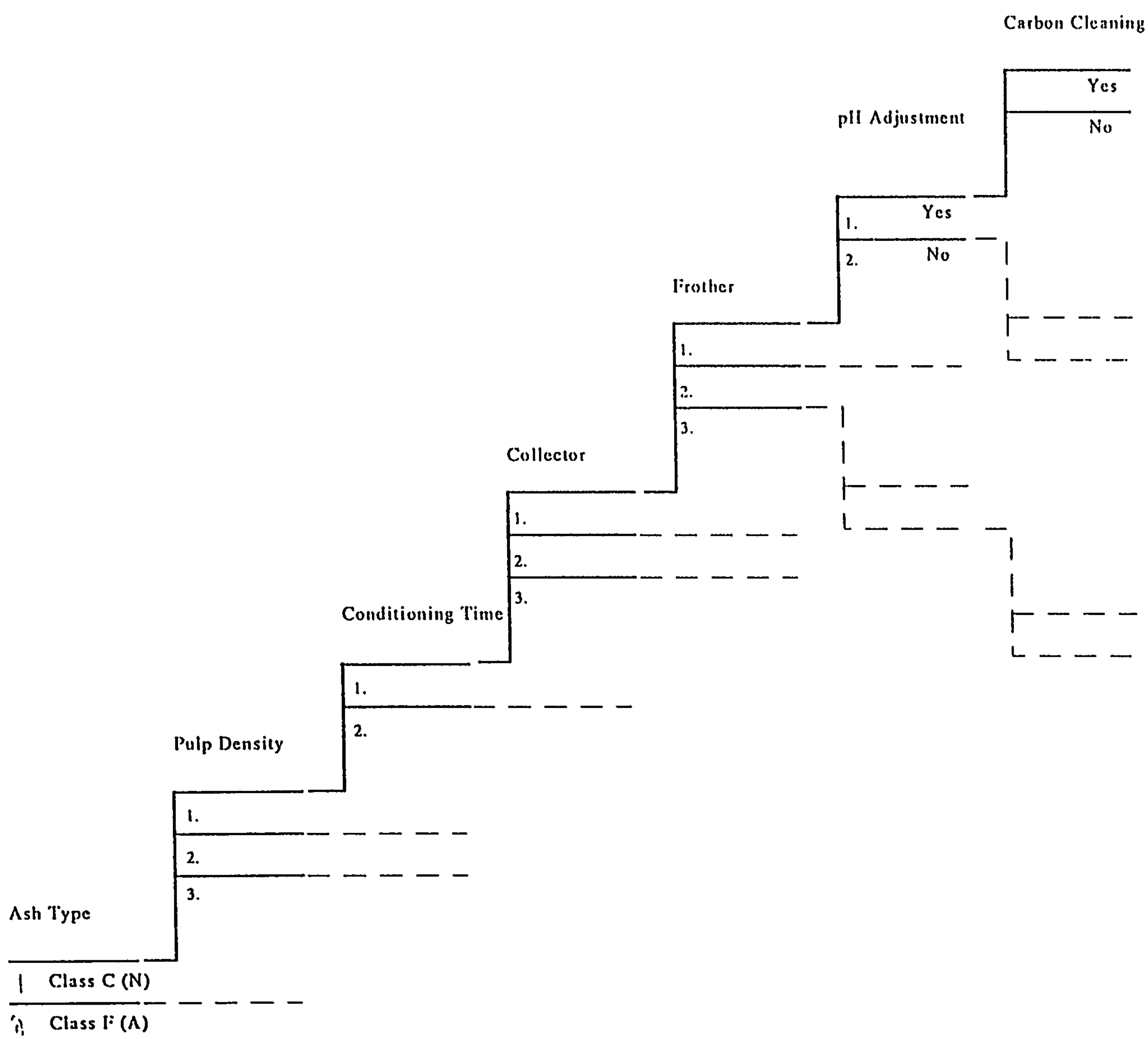
Flotation Test #	% Weight Cell	% Weight Froth	Carbon Cell	Carbon Froth
X.X.X.X.X.X				

Flotation Testing



Laboratory Flotation Testing

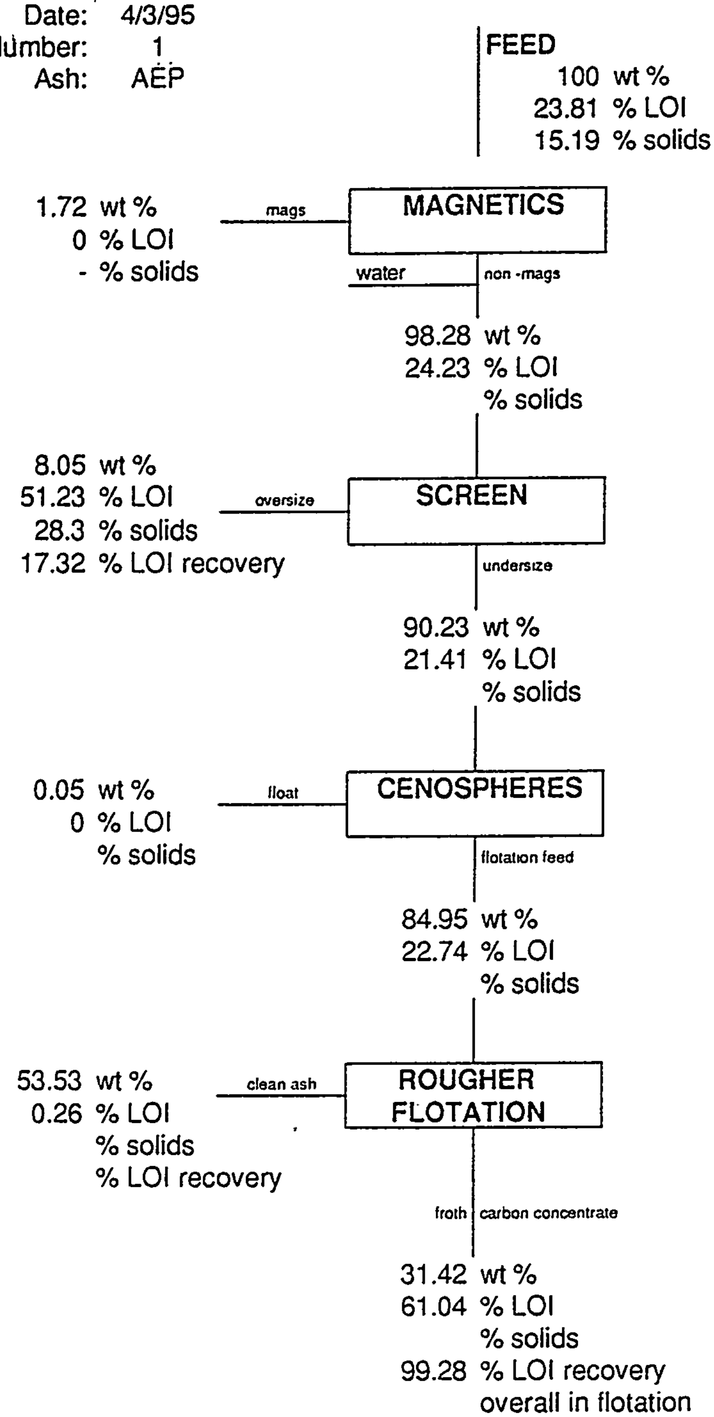
Seperation of Carbon From Ash Stream



APPENDIX C

Pilot Run Results

Project: E21670
Date: 4/3/95
Run Number: 1
Ash: AEP



NOTES:

flotation recovery calculated with LOI using flot feed, clean ash, and carbon concentrate

shakedown run

one stage of flotation

Project: E21670
Date: 4/12/95
Run Number: 2
Ash: AEP

NOTES:

flotation recovery calculated with LOI using flot feed, clean ash, and carbon concentrate

"measured" values were from weights of the overall sample collected.

Feed water @ 1.90 gpm

Screen moved to after cenosphere separation

Two stages of cleaner flotation

Carl Togni from AEP observed the run.

FEED
100 wt %
26.43 % LOI
17.33 % solids

MAGNETICS

mags
2.13 wt %
0 % LOI
- % solids

water

non -mags
97.87 wt %
27.01 % LOI
9.7 % solids

CENOSPHERES

float
0.05 wt %
0 % LOI
- % solids

sink
97.82 wt %
27.02 % LOI
9.7 % solids

SCREEN

oversize
11.60 wt %
50.51 % LOI
18.5 % solids
22.16 % LOI recovery

flotation feed
86.22 wt %
23.86 % LOI
8.57 % solids

wt %
61.3 % LOI
9.11 % solids

86.22 wt %
23.86 % LOI
% solids

ROUGHER FLOTATION

clean ash
52.83 % wt measured
59.07 wt %
2.71 % LOI
5.95 % solids

froth / cleaner feed
wt %
% LOI
% solids

CLEANER FLOTATION

clnr tails
wt %
% LOI
% solids

froth / 2nd cleaner feed
wt %
% LOI
% solids

2nd CLEANER FLOTATION

2nd clnr tails
wt %
53.39 % LOI
4.19 % solids

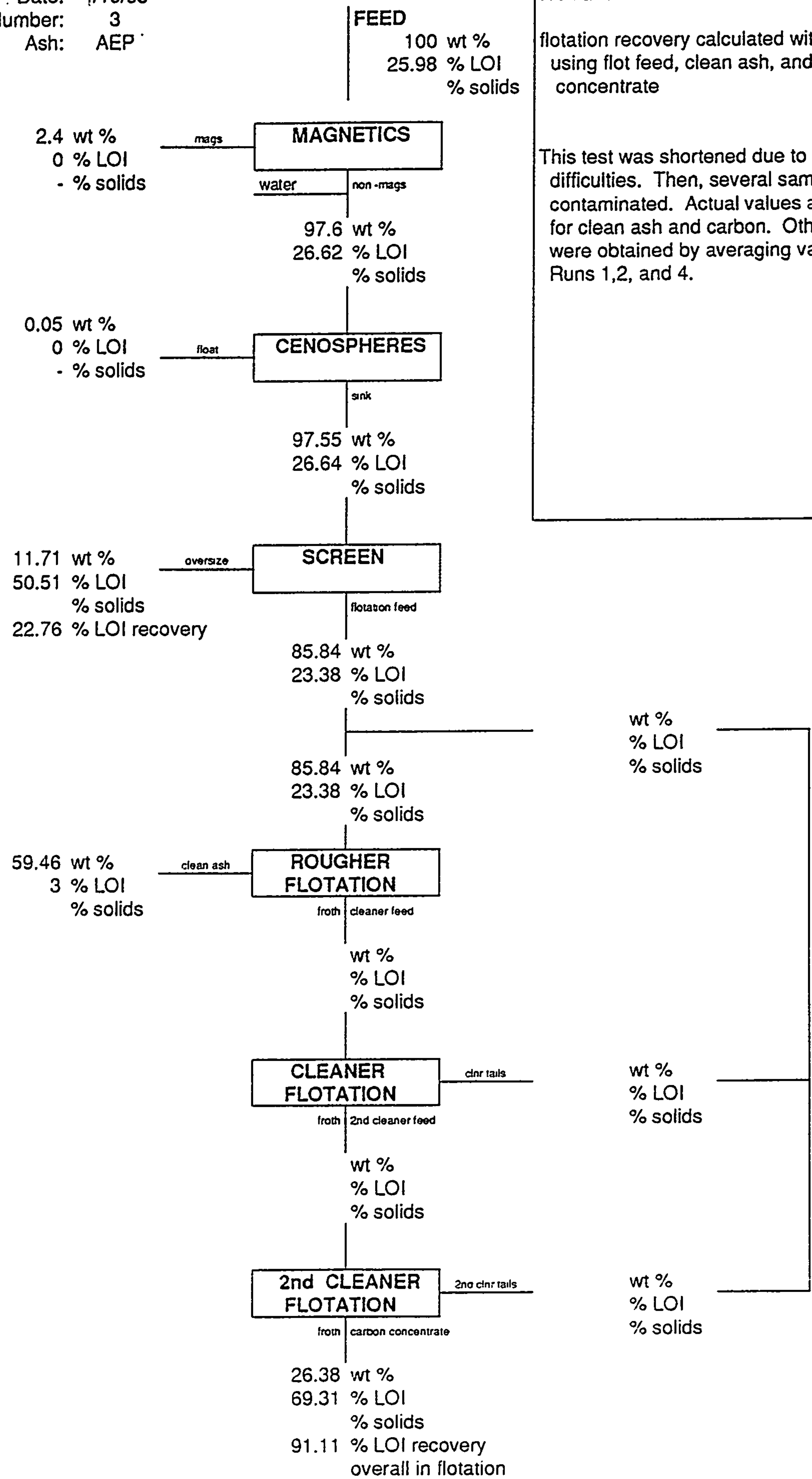
froth / carbon concentrate
27.15 wt %
21.06% wt measured
69.87 % LOI
11.08 % solids
92.22 % LOI recovery overall in flotation

Project: E21670
Date: 4/19/95
Run Number: 3
Ash: AEP

NOTES:

flotation recovery calculated with LOI using flot feed, clean ash, and carbon concentrate

This test was shortened due to mechanica difficulties. Then, several samples were contaminated. Actual values are used for clean ash and carbon. Other values were obtained by averaging values from Runs 1,2, and 4.

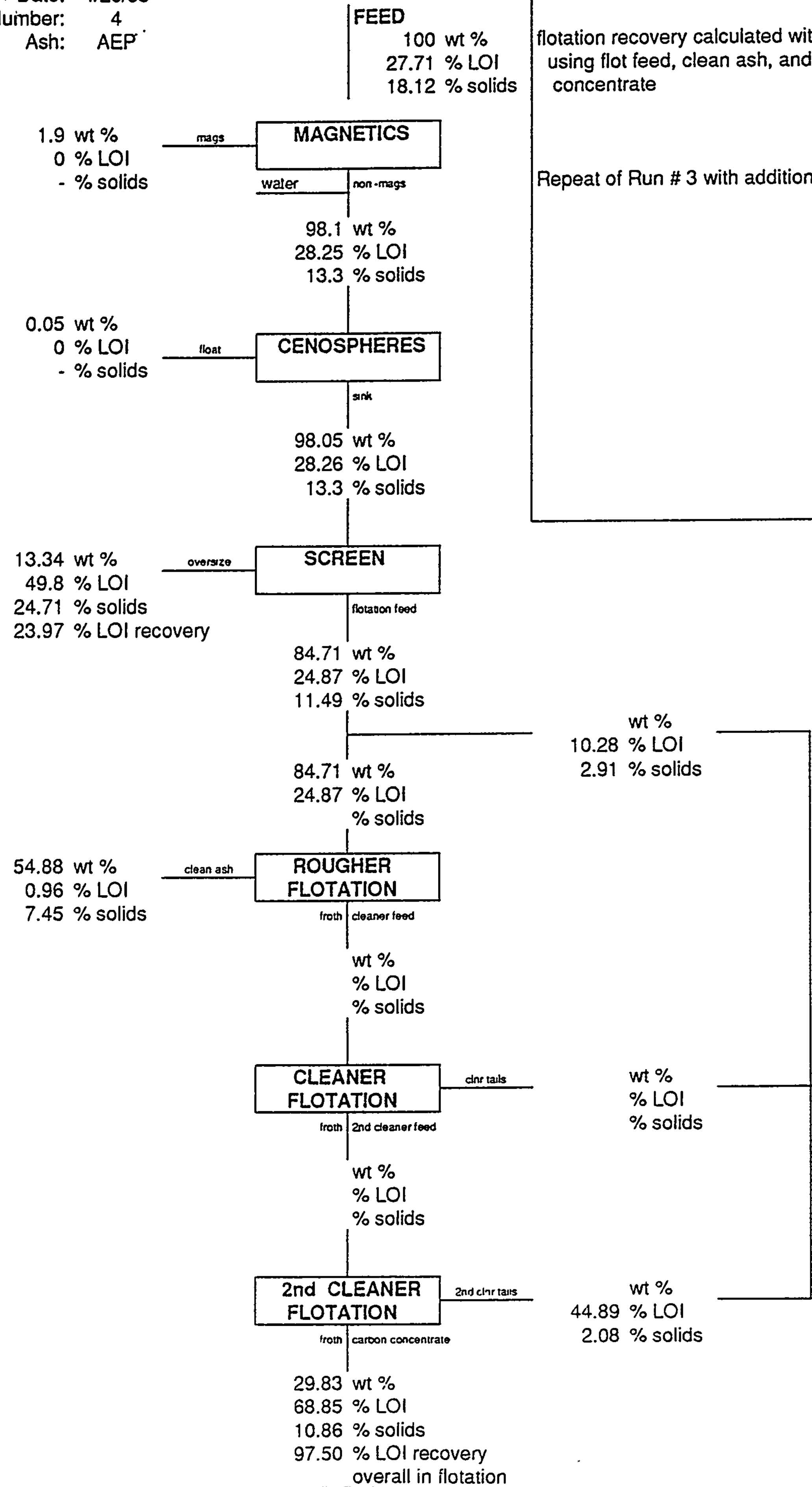


Project: E21670
Date: 4/20/95
Run Number: 4
Ash: AEP

NOTES:

flotation recovery calculated with LOI using flot feed, clean ash, and carbon concentrate

Repeat of Run # 3 with additional frother



22-Jun-95

Project: E21670
Date: 5/3/95
Run Number: 5
Ash: BGE

NOTES:

flotation recovery calculated with LOI using flot feed, clean ash, and carbon concentrate

FEED
100 wt %
6.39 % LOI
17.6 % solids

MAGNETICS

mags
1 wt %
0 % LOI
- % solids

water

non-mags
99 wt %
6.45 % LOI
12.9 % solids

CENOSPHERES

float
0.05 wt %
0 % LOI
- % solids

sink
98.95 wt %
6.46 % LOI
12.9 % solids

SCREEN

oversize
3.43 wt %
23.1 % LOI
21.2 % solids
12.40 % LOI recovery

flotation feed
95.52 wt %
5.86 % LOI
12.6 % solids

wt %
5.69 % LOI
6.04 % solids

95.52 wt %
5.86 % LOI
% solids

ROUGHER FLOTATION

clean ash
77.89 wt %
1.3 % LOI
9.75 % solids

froth | cleaner feed
wt %
% LOI
% solids

CLEANER FLOTATION

clnr tails
wt %
% LOI
% solids

froth | 2nd cleaner feed
wt %
% LOI
% solids

2nd CLEANER FLOTATION

2nd clnr tails
wt %
7.88 % LOI
4.65 % solids

froth | carbon concentrate
17.63 wt %
26.01 % LOI
13.8 % solids
81.91 % LOI recovery
overall in flotation

Project: E21670
Date: 5/31/95
Run Number: 6
Ash: NPC

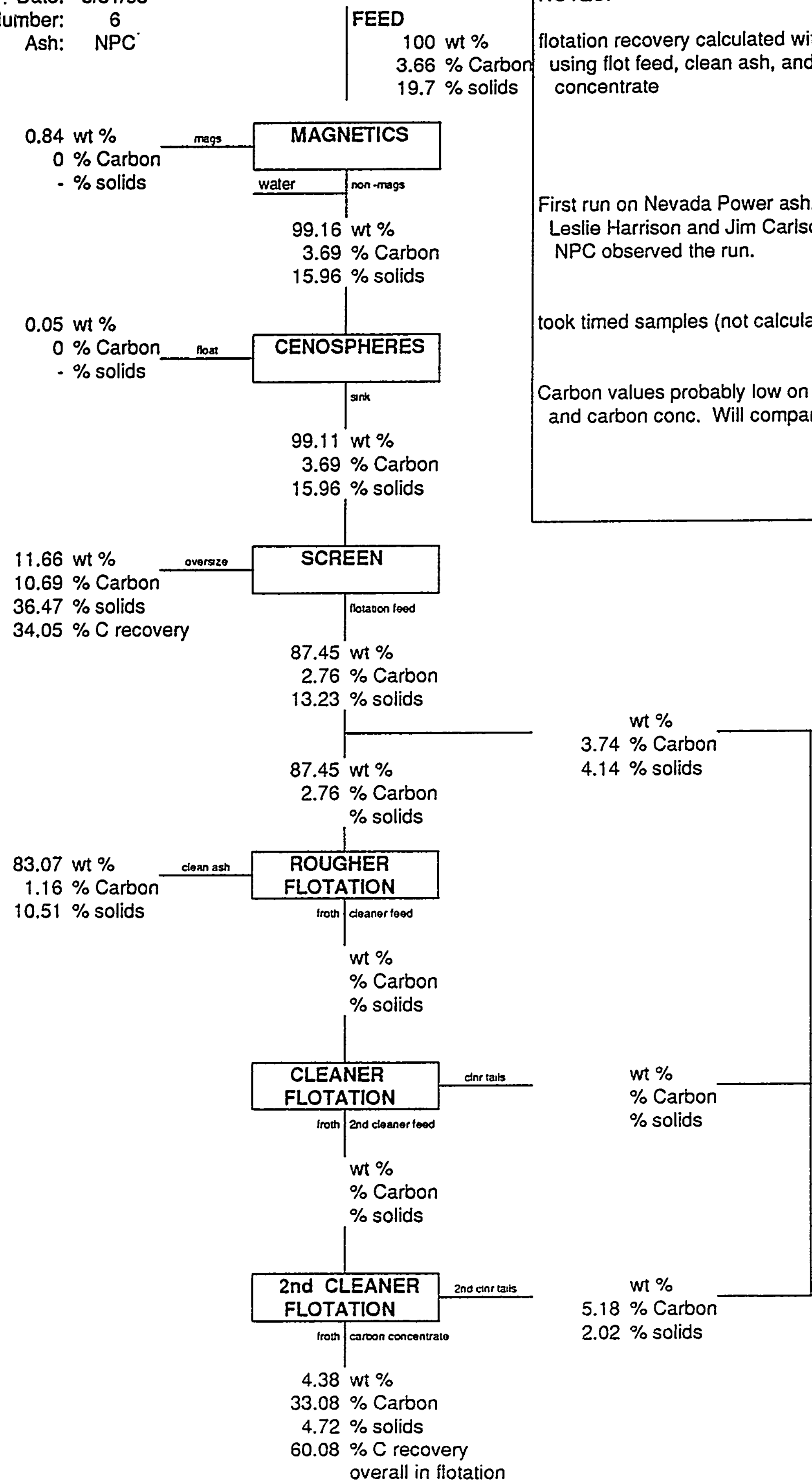
NOTES:

flotation recovery calculated with carbon using flot feed, clean ash, and carbon concentrate

First run on Nevada Power ash. Leslie Harrison and Jim Carlson from NPC observed the run.

took timed samples (not calculated yet)

Carbon values probably low on screen o's and carbon conc. Will compare w/ LOIs.



22-Jun-95

APPENDIX D

Concrete Testing Information

Table 1 - Summary of coarse aggregate grading limits and 6A aggregate for this study.				
Sieve Size	**ASTM C 33 Requirement wt% passing size # 57**	**6A aggregate MDOT* Requirement wt% passing**	**6A aggregate used in this study wt% passing**	**6A aggregate used in this study wt% retained**
1 inch	95-100	95-100	96	4
0.5 inch	25-60	30-60	36	60
No. 4 Mesh	0-10	0-8	3	
* MDOT: Michigan Department of Transportation				

Table 2 - Summary of fine-aggregate grading limits and properties of 2NS sand used in the present study.			
Sieve Size	**ASTM C 33 Requirement wt% passing**	**2NS sand (MDOT* Requirement) wt% passing**	**2NS sand used in this study wt% passing**
3/8" (9.5 mm)	100	100	100
No. 4 (4.74 mm)	95-100	95-100	99.0
No. 8 (2.36 mm)	80-100	65-95	83.5
No. 16 (1.18 mm)	50-85	35-75	63.7
No. 30 (0.6 mm)	25-60	20-55	46.2
No. 50 (0.3 mm)	10-30	10-30	22.1
No. 100 (0.15 mm)	2-10	0-10	9.2
Fineness modulus	2.3-3.1	2.50-3.35	2.76
Specific gravity	2.66		2.66
* MDOT: Michigan Department of Transportation			

Table 3 - Summary of AEP (Class F) Fly Ash Properties

Chemical Composition	As-received ash	Clean ash	ASTM C 618 (Class F ash)
SiO_2 (wt%)	44.0	58.6	
Al_2O_3 (wt%)	22.4	29.2	
Fe_2O_3 (wt%)	5.3	5.2	
Total $SiO_2+Al_2O_3+Fe_2O_3$, (wt%)	*71.7*	*93.0*	*70 (minimum)*
CaO (wt%)	0.76	0.85	
MgO (wt%)	0.86	1.11	
Na_2O (wt%)	0.32	0.42	
K_2O (wt%)	2.35	3.16	
TiO_2 (wt%)	1.11	1.33	
P_2O_5 (wt%)	0.03	0.09	
LOI (wt%)	21.7	0.40	6.0 (maximum)
SO_3 (wt%)	NA	NA	5.0 (maximum)
Total (wt%)	98.8	100.4	
Physical Test Results			
Moisture content (%)	0.25	0.20	3.0 (maximum)
325 mesh retained (%)	32.9	23.1	34 (maximum)
Specific gravity (g/cm^3)	2.13	2.19	

NA: Not currently available

Table 4 - Summary of BGE (Class F) Fly Ash Properties			
Chemical Composition	**As-received ash**	**Clean ash**	**ASTM C 618 (Class F ash)**
SiO_2 (wt%)	56.6	NA	
Al_2O_3 (wt%)	27.8	NA	
Fe_2O_3 (wt%)	3.3	NA	
Total $SiO_2+Al_2O_3+Fe_2O_3$, *(wt%)*	*87.7*	*NA*	*70 (minimum)*
CaO (wt%)	1.04	NA	
MgO (wt%)	0.80	NA	
Na_2O (wt%)	0.28	NA	
K_2O (wt%)	2.25	NA	
TiO_2 (wt%)	1.50	NA	
P_2O_5 (wt%)	0.01	NA	
LOI (wt%)	6.22	NA	6.0 (maximum)
SO_3 (wt%)	NA	NA	5.0 (maximum)
Total (wt%)	99.8	NA	
Physical Test Results			
Moisture content (%)	0.17	0.20	3.0 (maximum)
325 mesh retained (%)	19.5	NA	34 (maximum)
Specific gravity (g/cm^3)	2.24	2.20	

NA: Not currently available

Table 5 Summary of properties of dust collector ash (D.C. ash), from a previous project

Chemical Compositions (wt%)	**As-received ash**	**Cleaned ash**	**ASTM C 618 Class F ash**
SiO_2	44.00	57.00	
Al_2O_3	18.80	26.10	
Fe_2O_3	6.25	4.37	
Total $SiO_2+Al_2O_3+Fe_2O_3$	*69.05*	*87.47*	*70 (minimum)*
CaO	0.76	1.05	
MgO	0.44	0.67	
Na_2O	0.22	0.43	
K_2O	1.40	1.40	
TiO_2	0.77	1.14	
P_2O_5	0.28	0.42	
BaO	0.13	0.16	
MnO	0.05	0.05	
LOI (wt%)	29.82	2.53	6.0 (maximum)
SO_3 (wt%)	0.46	0.10	5.0 (maximum)
Total (wt%)	103.38	95.00	
Physical Test Results			
Moisture content (%)	0.55	0.35	3.0 (maximum)
325 mesh retained (%)	61.50	22.47	34 (maximum)
Specific gravity (g/cm^3)	2.24	2.30	

Table 6 - Summary of FA5 (Class F) Fly Ash Properties, from a previous project			
Chemical Composition	**As-received ash**	**Clean ash**	**ASTM C 618 (Class F ash)**
SiO_2 (wt%)	54.8	57.5	
Al_2O_3 (wt%)	28.8	30.2	
Fe_2O_3 (wt%)	2.5	3.2	
Total $SiO_2+Al_2O_3+Fe_2O_3$, (wt%)	*87.1*	*90.9*	*70 (minimum)*
CaO (wt%)	0.74	0.77	
MgO (wt%)	0.83	0.87	
Na_2O (wt%)	0.29	0.30	
K_2O (wt%)	2.70	2.80	
P_2O_5 (wt%)	0.16	0.17	
LOI (wt%)	7.25	0.61	6.0 (maximum)
SO_3 (wt%)	0.31	0.32	5.0 (maximum)
Total (wt%)	99.11	96.42	
Physical Test Results			
Moisture content (%)	0.28	0.13	3.0 (maximum)
325 mesh retained (%)	9.01	4.91	34 (maximum)
Specific gravity (g/cm^3)	2.22	2.22	

Table 7 - Effect of amount of cement replaced by as-received AEP fly ash on properties of 35S concrete (lb per cubic yard)*.				
Amount replaced	**0% Mix1**	**8% Mix2**	**20%**	**30%**
W/C ratio**	0.50	0.500	0.510	0.520
Cement, lb	564	517	451	395
Fly ash, lb	0	78	141	212
Water, lb	297	312	316	329
Fine aggregate MC<0.5%	1150	1050	1014	939
Coarse aggregate MC<1.0%	1845	1845	1845	1845
Water Reducer, (oz)	0	0	0	0
AEA#, oz	9.9	10.7	10.9	11.1
Slump, in	3.5	3.2	2.5	2.2
Air, (%)	7.1	2.0	1.8	1.9
pcf##	148.8	151.2	149.6	148.9
7 day strength, (psi)@	3692	4223	3357	2420
28 day strength, (psi)@	4676	5801	4582	3681
91 day strength, (psi)@	5512	NA	NA	NA

Notes:

*	Specified design strength is 3500 psi.
**	W/C ratio = water/(cement + fly ash) ratio.
#	Grace Daravair air entraining agent was used.
##	Fresh unit weight (lb per cubic yard)
@	Compressive strength, psi, average of 3 data
NA	Not currently available

Table 8 - Effect of amount of cement replaced by as-received FA-5 fly ash on properties of 35S concrete (lb per cubic yard)*. Data from a previous project.					
Amount replaced	**0%, Mix1**	**8%, Mix2**	**20%**	**30%**	**40%**
W/C ratio**	0.50	0.475	0.475	0.48	0.472
Cement, lb	564	517	451	395	338
Fly ash, lb	0	78	141	212	282
Water, lb	296.5	297.0	295.6	305.2	306.8
Fine aggregate MC<0.5%	1150	1094	1076	1015	973
Coarse aggregate MC<1.0%	1845	1845	18245	1845	1845
AEA#, oz	9.9	10.6	10.9	11.0	11.4
Slump, in	3.6	2.5	2.5	3.0	2.9
Air, (%)	7.8	3.9	2.2	1.6	1.6
pcf##	145.4	148.0	150.8	150.1	148.0
7 day strength, (psi)@	3550	4438	3558	3028	2418
28 day strength, (psi)@	4646	4882	4900	4508	3878
91 day strength, (psi)@	5006	6101	5807	5548	5012

NOTES

*	Specified design strength is 3500 psi.
**	W/C ratio = water/(cement + fly ash) ratio.
#	Grace Daravair air entraining agent was used.
##	Fresh unit weight (lb per cubic yard)
@	Compressive strength, psi, average of 3 data

Table 9 - Effect of amount of cement replaced by cleaned AEP fly ash on properties of 35S concrete (lb per cubic yard)*.					
Amount replaced	**0% Mix1**	**8% Mix2**	**20%**	**30%**	**20%**
W/C ratio**	0.50	0.465	0.460	0.440	0.425
Cement, lb	564	517	451	395	451
Fly ash, lb	0	78	141	212	141
Water, lb	297	291	287	281	266
Fine aggregate MC<0.5%	1150	1093	1098	1075	1155
Coarse aggregate MC<1.0%	1845	1845	1845	1845	1845
Water Reducer, oz	0	0	0	0	17.8
AEA#, oz	9.9	10.6	10.9	11.1	10.9
Slump, inch	3.5	3.2	3.0	3.5	3.0
Air (%)	7.1	6.5	5.5	6.9	6.5
pcf##	148.8	145.4	145.8	144.2	146.6
7 day strength (psi)@	3692	3263	2561	2273	3103
28 day strength (psi)@	4676	4446	3999	3681	4105
91 day strength (psi)@	5512	NA	NA	NA	NA

NOTES

*	Specified design strength is 3500 psi.
**	W/C ratio = water/(cement + fly ash) ratio.
#	Grace Daravair air entraining agent was used.
##	Fresh unit weight (lb per cubic yard)
@	Compressive strength, psi, average of 3 data
NA	Not currently available

Table 10 - Effect of amount of cement replaced by cleaned FA-5 fly ash on properties of 35S concrete (lb per cubic yard)*. Data from a previous project.						
Amount replaced	**0% Mix1**	**8% Mix2**	**20%**	**30%**	**40%**	**20%**
W/C ratio**	0.50	0.475	0.465	0.465	0.465	0.430
Cement, lb	564	517	451	395	338	451
Fly ash, lb	0	78	141	212	282	141
Water, lb	296	294	290	293	302	270
Fine aggregate MC<0.5%	1150	1103	1093	1046	984	1148
Coarse aggregate MC<1.0%	1845	1845	1845	1845	1845	1845
Water Reducer, oz	0	0	0	0	0	17.8
AEA#, oz	9.9	10.6	10.9	11.1	11.4	10.9
Slump, inch	3.6	3.5	3.7	3.5	2.8	3.8
Air (%)	7.8	6.5	7.0	4.8	4.0	6.2
pcf##	145.4	142.7	145.1	144.7	146.0	146.8
7 day strength, (psi)@	3550	3719	3017	2698	2239	3437
28 day strength, (psi)@	4646	4152	3869	4022	3909	4785
91 day strength, (psi)@	5006	5012	4930	4501	4493	5913

NOTES

- * Specified design strength is 3500 psi.
- ** W/C ratio = water/(cement + fly ash) ratio.
- \# Grace Daravair air entraining agent was used.
- \## Fresh unit weight (lb per cubic yard)
- @ Compressive strength, psi, average of 3 data

Table 11- Effect of amount of cement replaced by as-received BGE fly ash on properties of 35S concrete (lb per cubic yard)*.					
Amount replaced	**0% Mix1**	**8% Mix2**	**20%**	**30**	**40%**
W/C ratio**	0.50	0.475	0.475	0.475	0.475
Cement, lb	564	517	451	395	338
Fly ash, lb	0	78	141	212	282
Water, lb	297	297	299	302	309
Fine aggregate MC<0.5%	1150	1095	1078	1025	971
Coarse aggregate MC<1.0%	1845	1845	1845	1845	1845
Water Reducer, (oz)	0	0	0	0	0
AEA#, oz	9.9	10.7	10.9	11.1	11.4
Slump, in	3.5	3.5	2.2	3.0	3.0
Air, (%)	7.1	1.8	1.8	1.5	2.0
pcf##	148.8	152.2	151.5	151.3	149.4
7 day strength, (psi)@	3692	4482	3852	2968	2214
28 day strength, (psi)@	4676	6054	5383	4429	3634
91 day strength, (psi)@	5512	7173	6749	5899	4929

NOTES

*	Specified design strength is 3500 psi.
**	W/C ratio = water/(cement + fly ash) ratio.
#	Grace Daravair air entraining agent was used.
##	Fresh unit weight (lb per cubic yard)
@	Compressive strength, psi, average of 3 data
NA	Not currently available

Table 12 Effect of amount of cement replaced by cleaned BGE fly ash on properties of 35S concrete (lb per cubic yard)*.

Amount replaced	0% Mix1	8% Mix2	20%	30%	40%
W/C ratio**	0.50	0.465	0.460	0.460	0.455
Cement, lb	564	517	451	395	338
Fly ash, lb	0	78	141	212	282
Water, lb	297	291	287	293	296
Fine aggregate MC<0.5%	1150	1110	1098	1045	1000
Coarse aggregate MC<1.0%	1845	1845	1845	1845	1845
Water Reducer, (oz)	0	0	0	0	0
AEA#, oz	9.9	10.7	10.9	11.1	11.4
Slump, in	3.5	3.2	2.2	3.0	4.0
Air, (%)	7.1	5.0	4.5	3.2	4.5
pcf##	148.8	147.4	148.3	147.8	145.1
7 day strength, (psi)@	3692	3322	2680	NA	NA
28 day strength, (psi)@	4676	4588	NA	NA	NA
91 day strength, (psi)@	5512	NA	NA	NA	NA

NOTES

*	Specified design strength is 3500 psi.
**	W/C ratio = water/(cement + fly ash) ratio.
#	Grace Daravair air entraining agent was used.
##	Fresh unit weight (lb per cubic yard)
@	Compressive strength, psi, average of 3 data
NA	Not currently available